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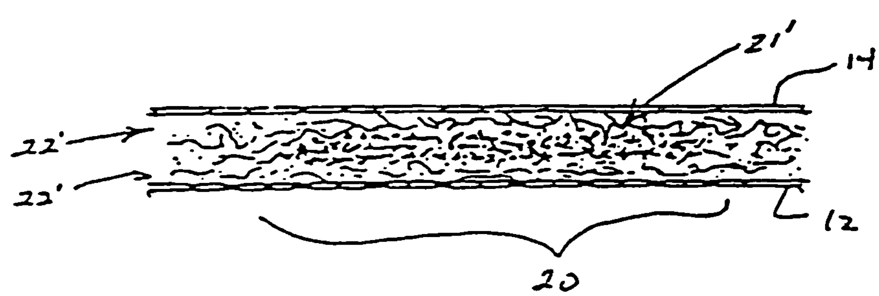
(54) Title: ABSORBENT ARTICLES CONTAINING HIGH FVAUL SAP



(57) Abstract: A disposable absorbent product (10) that comprises a liquid permeable top sheet (14); a liquid impermeable back sheet (12); and a two phase absorbent panel (16) positioned between the top sheet and the back sheet provided. The two phase absorbent panel structure comprises a wood pulp fiber and a surface cross linked superabsorbent material. In at least one layer of the two phase absorbent matrix, the superabsorbent material comprises substantially a continuous phase of the matrix (21'), where a sufficient quantity of particles of the superabsorbent material are in contact with each other to thereby define a capillary network for facilitating liquid transport within the matrix.

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ABSORBENT ARTICLES CONTAINING HIGH FVAUL SAP

Field of Invention

The present invention relates generally to absorbent materials that are used in disposable articles such as diapers, incontinence products and catamenial products. In particular the present invention is directed to composite absorbent materials comprising superabsorbent polymeric materials having a high fluid capacity with minimal gel blocking properties. The present invention also relates to a method and apparatus for predicting absorbency performance of such composite absorbent materials.

Background of the Invention

Absorbent composite materials comprising superabsorbent polymeric materials that can absorb large amounts of liquids, such as water or body exudates, have many applications in disposable absorbent articles such as baby diapers, sanitary napkins, wound dressings, bandages, incontinent pads, and the like.

Preferably, the absorbent composites should absorb and retain large volumes of liquids under moderate pressures. For instance, absorbent composites incorporated in a disposable baby diaper should quickly absorb and retain urine without leakage even when a baby is exerting a load to the diaper by sitting on in. Moreover, a baby diaper also needs [to] be able to absorb the total volume that results from multiple insults.

Historically, diapers and other such disposable absorbent articles have been made of comminuted wood pulp fibers (also called wood pulp fluff) or other hydrophilic fibrous materials in which a superabsorbent material has been dispersed. For example, U.S. Patent No. 5,330,822 issued to Berg et al., U.S. Patent No.

5,147,343 issued to Kellenberger, U.S. Patent No. 4,673,402 issued to Weisman, U.S. Patent No. 5,281,207 issued to Chmielewski et al., and U.S. Patent No. 4,834,735 issued to Alemany et al. disclose absorbent composites of the type comprising a porous matrix of fibers and a superabsorbent polymer dispersed among the fibers. Superabsorbent polymers or materials are also known under the more technical term ionic hydrocolloids. Generally, a superabsorbent polymer should absorb or imbibe at least about 10 times its own weight of fluid and retain it under moderate pressures. These aforementioned patents disclose many types of SAPs and methods for making them, and are incorporated herein by reference for all purposes. However, this absorbent composite system has drawbacks. For example, it seems intuitive that in order to increase the fluid capacity of the absorbent composite, i.e., the amount of liquid that the absorbent composite could absorb, one might simply increase the amount of superabsorbent material in the absorbent particle. However, as the amount of superabsorbent material in the absorbent composite is increased, a point of "diminishing returns" is seen; that is, the absorptivity or fluid capacity of the absorbent article does not increase proportionally with the amount of SAP incorporated into the composite. Thus, increased absorption may not be obtained by merely increasing the amount of superabsorbent material in the absorbent composite. One explanation for this problem is a phenomenon called gel blocking.

Generally, it is believed that a liquid is transported within an absorbent structure comprising wood pulp fiber and a superabsorbent material by the wicking action of the wood pulp fibers and by capillary transport, which relies on the interstitial free space or free volume between the particulate superabsorbent materials and the wood pulp fibers. As more superabsorbent material is dispersed in the fiber

matrix of prior art absorbent composites at certain points, the swelling of the superabsorbent typically results in the blocking of the capillaries, thus preventing the liquid to reach SAP particles that haven't been fully saturated with liquid.

It is believed by the inventor of the present invention that when the swollen SAP particles are in contact as a result of the amount of SAP added in the composite, their surfaces may deform or coalesce to form a generally continuous mass or gel, which reduces the free space available for liquid transport via capillary action. As a result, the capillary transport of a liquid to superabsorbent material that has not absorbed a liquid is reduced. The gel formed by the particles that have absorbed a liquid and have swelled blocks the transport of additional liquid to superabsorbent materials that have not absorbed the liquid. Generally, because of this gel blocking action, prior art absorbent composites with SAP content greater than about 600 percent by weight of the total composite SAP and fiber weight have not been effective. Typically, prior art absorbent composites are limited to structures where the SAP material forms distinct particles inside the continuous fiber matrix phase.

Summary of the Invention

The present invention overcomes the aforementioned limitations of the prior art. One object of the present invention is to provide an absorbent composite that has a high fluid capacity, also referred to as absorptive capacity, with minimal gel blocking properties. Particularly, the present invention is directed to composite absorbent materials comprising a surface crosslinked, superabsorbent polymer. It has been discovered that the use of SC-SAPs allows to significantly increase the amount of superabsorbent in the absorbent composite without the gel blocking phenomenon that marred the fluid capacity of prior art absorbent composites. For brevity, a

superabsorbent polymer will be referred herein after as SAP and a surface crosslinked SAP will be referred to as SC-SAP. The SC-SAP is generally dispersed within a matrix of wettable fibers such wood pulp (fluff), cotton linters, synthetic fibers and mixtures thereof. The absorbent composites of the present invention contain at least a layer wherein the SC-SAP forms a substantially continuous phase, that is a substantial number of the SC-SAP particulates are in contact with each other in the dry state, prior to absorption of any liquid. Preferably, the SC-SAP should form a substantially continuous phase throughout the absorbent composite material, since this structure maximizes the amount of SAP incorporated in the composite, however, the FVAUL free volume of the composite which is described in the following description should be at least about 15 percent. Put it simply, FVAUL is a measure of the absorbent's ability to maintain open a sufficient number of capillaries in the absorbent composite matrix.

Another object of the present is to provide a method and apparatus for predicting the fluid capacity performance of such composite absorbent materials. In particular, the present invention provides a method and apparatus that are particularly suited for predicting the gel blocking tendency of such absorbents. In its broadest aspects the present invention method comprises measuring the free volume of an absorbent composite after contacting the absorbent composite with a liquid solution, under load and for a specified period of time. It has been discovered that the free volume is a reliable predictor of the performance of such absorbent composites including absorptive capacity, in-use leakage, and gel blocking properties. Generally, absorbent components with high free volume values are free of the gel blocking phenomena observed with prior art absorbent composites. A preferred embodiment of

the present invention method also referred to hereinafter as finite volume absorbency under load (FVAUL) comprises contacting an absorbent composite with a 1.0 weight percent solution of sodium chloride in water, under a 1.6 psi load, for 600 seconds and calculating the free volume of the absorbent composite. The free volume value obtained under these conditions will be referred hereinafter as the "FVAUL free volume" or "FVAUL property" or "FVAUL value". Of course, it should be understood that many variations of the present invention method can be made by a skilled artisan without departing from the scope of the present invention.

It is still another object of the present invention to provide an apparatus for measuring the FVAUL property of an absorbent composite. It is further another object of the present invention to provide improved disposable absorbent articles such as but not limited to diapers, sanitary napkins that incorporate the absorbent composite materials of the present invention. It is also another object of the present invention to provide a method for absorbing spilled liquids from surfaces by employing the absorbent articles of the present invention. These and other objects of the present invention will become apparent to those skilled in the art from the following description. Finally, it should be understood that many other objects and variations of the present invention absorbent composites, methods and apparatuses may be easily developed by those skilled in the art upon reading the following description of specific embodiments of the present invention without departing from the intended scope of the present invention.

Brief Description of the Drawings

While the specification concludes with claims which particularly point out and distinctly claim the subject matter of the present invention, it is believed that the

invention will be better understood from the following description taken in conjunction with the accompanying drawings in which:

FIGURE 1 is a pictorial representation of a cross-sectional portion of an absorbent composite of the present invention;

FIGURE 2 is a perspective view, partially cutaway, of a disposable absorbent diaper of the present invention.

FIGURE 2A is a vertical cross-sectional view of a target region 21' of the diaper shown in FIGURE 2;

FIGURE 3 is a diagrammatic view of a preferred embodiment of an apparatus used in an FVAUL method;

FIGURE 4 shows a close up view of the weight used in the FVAUL method;

FIGURES 5 and 6 are graphical presentations of the FVAUL test data. Specifically, Figure 5 shows the data obtained in Test No. 1 of Table 2, and Figure 6 shows the data obtained from Test No. 5 of Table 1.

FIGURES 7 and 8 are photomicrographs of specific embodiments of absorbent composites of the present invention.

FIGURE 9 is a photomicrograph of a prior art absorbent composite.

Detailed Description of the Present Invention

The present invention provides an absorbent composite that has a high fluid capacity, or absorptive capacity with minimal gel blocking properties. Particularly, the present invention is directed to a composite absorbent material comprising a continuous SC-SAP phase and fibers dispersed within the interstitial space between the SC-SAP particles. SC-SAPs and methods of making them are described in U.S. Patent Nos. 4,666,983 and 4,734,478 issued to Tsubakimoto et al. and which are

incorporated herein by reference for all purposes. Methods of preparing the absorbent composite structure are disclosed in U.S. Patent No.5,281,207 to Chmielewski, et al., which is incorporated herein by reference for all purposes. The SAP is combined with the wettable fibers in an amount from about 70% to about 95% by weight based on the combined weight of fibers and SAP by means suitable to distribute the SAP therein trying to form a substantially continuous phase of SAP. Generally, it is desirable to place the SAP somewhat evenly distributed throughout the absorbent composite. Any SAP known in the art which absorbs large amounts of liquid and can be surface cross-linked is suitable for use in the absorbent layer of the present invention. Also mixtures of more than one SC-SAP materials may be used. The SC-SAP is generally dispersed within a matrix of wettable fibers such as wood pulp (fluff), cotton linters, synthetic fibers and mixtures thereof, preferably wood pulp fluff or a mixture of wood pulp and synthetic fibers. Suitable synthetic fibers include polyethylene, polypropylene, polyesters, copolymers of polyesters and polyamides, and the like. Examples of preferred wood pulps that may be used in the practice of the present invention are provided in U.S. pat. No. 5,147,343 which is incorporated herein by reference for all purposes. The fibers are generally hydrophilic or are rendered hydrophilic through a surface treatment.

Typically, the absorbent composite of the present invention comprises a two phase matrix containing a first fibrous phase and a second SC-SAP phase. The SC-SAP phase, preferably comprises particulate, flaked, or fibrous SC-SAP material, preferably particulate SC-SAP material. The SC-SAP material should preferably form a substantially continuous phase meaning that there is sufficient number of SC-SAP particles in the absorbent composite so that a substantial number of the SC-SAP

particulates are in contact with each other in the dry state, prior to absorption of any liquid. Preferably, the SC-SAP phase should be continuous throughout the absorbent composite structure of the present invention since this configuration allows for the maximum amount of SC-SAP material to be incorporated in the absorbent composite and since, generally, the greater the amount of SC-SAP material in the composite the greater the fluid capacity of the composite will be. It has been generally found that for a continuous SC-SAP phase to exist the SC-SAP material should constitute at least 70 percent by weight of the weight of the continuous phase SC-SAP layer. Preferably, the SC-SAP should constitute at least 80 percent by weight, and more preferably at least 90 percent by weight.

U.S. Pat. No. 5,147,343 issued to Kellenberger, U.S. Pat. No. 4,673,402 issued to Weisman, U.S. Pat No. 5,281,207 issued to Chmielewski et al., and U.S. Patent No. 4,834,735 issued to Alemany, et al. disclose many types of SAPs and methods for making them, and are incorporated herein by reference for all purposes and in a manner that is consistent herewith. SC-SAPs and methods of making them are described in U.S. Patent Nos. 4,666,983 and 4,734,478 issued to Tsubakimoto et al. which are incorporated herein by reference for all purposes and. in a manner that is consistent herewith. Also, U.S. Pat. No. 5,281,207 to Chmielewski, et al. generally discloses methods and materials for making an absorbent article and is also incorporated herein by reference for all purposes and in a manner that is consistent herewith.

A sufficiently small quantity of wood pulp fibers should be included in order to, inter alia, maintain the stability of the adsorbent structure and preserve the wicking function of the fibers. The amount of the fibrous phase should range between about 5

percent by weight and 30 percent by weight of the total weight of the continuous SC-SAP phase absorbent composite, preferably, the fibers should not exceed 15 percent, and more preferably should not exceed 10 percent, by weight of the total weight of the absorbent. Moreover, in order to preclude the gel blocking phenomenon the FVAUL free volume of the continuous SC-SAP layer should be at least 15 percent at 600 seconds. It has been unexpectedly found that when the FVAUL free volume value is at least 15 percent then the absorbent material does not exhibit gel blocking at high SAP concentrations. Preferably, the FVAUL free volume of the continuous SC-SAP layer should be at least 20 percent, and more preferably at least 25 percent. Finally, the FVAUL free volume of absorbent composite of the present invention should not exceed 70 percent, preferably 60 percent, and most preferably 50 percent.

Figure 1 is a pictorial cross sectional representation of a portion of an absorbent composite material of the present invention. The diagram illustrates a continuous SC-SAP phase formed by the SC-SAP particles 21 and fibrous material 22 filling the interstitial space between the SC-SAP particles 21.

Albeit, Figure 1 shows that essentially all particles are connected with each other, it should be clear from the above description that the present invention does not require that all the particles are connected to one another but rather that a substantially continuous SC-SAP phase is formed. However, it is preferred that essentially all SC-SAP particles by connected to each other since such a structure would allow for optimizing fluid capacity provided, however, that the FVAUL free volume is kept above 15 percent.

Figures 7 and 8 are actual photomicrographs(magnification 50x) of absorbent composites of the present invention. Figures 7 and 8 show SC-SAP particles forming

what is characterized as a substantially continuous phase. This is to be contrasted with the photomicrograph of a prior art absorbent composite wherein the fibers are in a continuous phase while the SAP material is in the form of discrete particles that are not connected to each other.

The present invention also provides improved disposable absorbent articles such as but not limited to diapers, sanitary napkins that incorporate the absorbent composite of the present invention. Disposable diaper articles are described in U.S. Patent Nos. 4,673,402; 5,147,343; 5,330,822; 4,834,735; and 5,281,207, which are incorporated herein by reference for all purposes. A preferred disposable diaper, for the purpose of this invention, is shown in Figures 2 and 2A. In accordance with Figures 2 and 2A, a disposable diaper 10 comprises a liquid impermeable back sheet 12, a liquid permeable top sheet 14 and an absorbent panel structure 16 positioned between the top sheet 14 and the back sheet 12.

In accordance with the present invention, in at least a layer of the absorbent panel, in a target region thereof indicated by circle 21', taken in the Z-direction thereof (i.e., in a direction from top to bottom, away from the wearer), the superabsorbent material comprises a substantially continuous phase of the matrix. For purposes of this disclosure, the substantially continuous phase is provided wherein a sufficient quantity of particles of the superabsorbent material are in multiple point contact with each other, both prior to sorption of liquid and thereafter, to thereby define a capillary network for facilitating liquid transport within the panel structure. A sufficiently small quantity of wood pulp fibers, preferably at least about 5 percent and no more than about 30 percent on a weight percentage basis, are intermixed with the superabsorbent material in the continuous phase. This quantity of wood pulp fiber

acts to maintain the stability of the absorbent structure by integrating the region of the continuous phase of superabsorbent particles with adjacent portions of the absorbent structure. As a result, the target region of the absorbent panel structure, designated 20 in FIGURE 2, and which includes said layer, exhibits a free volume, at 600 seconds, of at least about 15 percent during finite volume absorbency under load (FVAUL) testing. The target region corresponds to the second and third fifths of the absorbent structure, measured from the front thereof.

As shown in the cross-sectional view FIGURE 2a, the layer of the absorbent matrix having the continuous phase portion 21' is preferably positioned between two layers, designated 22', each comprising predominantly wood pulp fibers. These layers 22' each comprise at least 80 percent and preferably as much as 95 percent or more, by weight basis, of wood pulp fibers. In the case of a diaper, the liquid permeable top sheet 14 allows urine to flow through the sheet to the absorbent panel structure 16 and also keeps the baby from directly contacting the absorbent panel structure. This configuration provides more comfort for the baby and also helps to position the absorbent panel structure. Liquid permeable top sheets, and liquid impermeable back sheets, are well known to those skilled in the art, and these components can be suitably selected in practicing the present invention.

Back sheet 12 is impermeable to liquids, and thus, helps to retain a liquid so that the liquid may be absorbed and retained by the absorbent panel structure. In a baby diaper, the impermeable back sheet is typically a sheet of plastic film, such as polyethylene, that helps to retain the urine so that the urine may be absorbed by the absorbent panel structure of the diaper. For a detailed discussion of materials that can

be used in the top and back sheet of a diaper, see U.S. Patent No. 5,281,207 issued to Chmielewski, et al. and which is incorporated herein for all purposes.

The absorbent panel structure 16 is made of a two phase matrix comprising wood pulp fiber and surface crosslinked polymeric superabsorbent material. As noted above, by two phase, it is meant that the absorbent panel structure has two components, fibers (preferably wood pulp) and a superabsorbent material. The absorbent structure may comprise more than one layer. For example, the absorbent structure may have a layer that is substantially wood pulp fiber, while on top of this layer the absorbent structure may have another layer of wood pulp fiber that contains particulate superabsorbent material dispersed in the wood pulp fiber. It is contemplated that many different combinations of layers may be used in the practice of the present invention. For example, in a preferred embodiment of the invention, in at least the target region 20, a three layer system is formed in which a layer containing superabsorbent particulate material in a substantially continuous phase is positioned between adjacent layers formed predominantly of wood pulp fiber.

A greater quantity of superabsorbent material is preferably found dispersed in the wood pulp fibers in the target region 20 than in any other portion of the absorbent panel structure. In the target region 20, in at least one layer thereof, the superabsorbent material comprises a substantially continuous phase. By continuous phase, it is meant that the quantity of superabsorbent particles is so great in the region as to contact each other and to thereby define a capillary network for facilitating liquid transport within the panel structure. Thus, in the continuous phase there is more superabsorbent particles than wood pulp fibers, preferably at least 70 percent up to about 90 percent, on a weight percentage basis of superabsorbent particles. However,

it should be noted that even in the continuous phase, the superabsorbent material is dispersed in the wood pulp fiber. A relatively small quantity of wood pulp fibers intermixed with the superabsorbent material is present in the continuous phase, for stability, since this small quantity of wood pulp fibers acts to integrate the continuous phase portion 21' with adjacent portions of the absorbent structure.

The continuous phase portion 21' containing superabsorbent particulate material may be substantially continuous across the entire width and length of the absorbent structure. The continuous phase portion 21' containing superabsorbent material is preferably located in specific targeted areas within the absorbent structure, such a target region 20, extended along a longitudinal centerline of the absorbent structure for at least the second and third fifths of the length of the absorbent structure. The continuous phase portion 21' can extend outwardly from the longitudinal centerline toward the side marginal edges of the article at least 20 percent-100 percent of the width of the absorbent structure, and preferably about 50 percent-70 percent. Because superabsorbent material is one of the most costly components of an absorbent structure, efficient use and positioning of the material is beneficial. Specific positioning of the superabsorbent material in areas most likely to be insulted with urine allows for the most cost effective utilization of this component. Specific positioning of superabsorbent material can be accomplished through any of several methods, such as by the method and apparatus as described and claimed in U.S. Patent 5,279,854, which is incorporated herein by reference. This specific positioning creates a target region 20 shown in FIGURE 2.

As noted, FIGURE 1 is a pictorial representation of a portion of the absorbent matrix wherein the substantially continuous phase comprises particulate

superabsorbent material 21, wood pulp fibers 22, and interstitial voids or free volume 23. As FIGURE 1 shows, the particles of superabsorbent material touch and the interstitial voids of spaces between the particles and fibers is called the free volume. The free volume is important as the free volume space is necessary to maintaining a capillary structure through which the liquid can be transported and stored. Thus, the amount of free volume in an absorbent structure is important to the absorbency characteristics of the continuous phase. The continuous phase may be described as a region of the absorbent structure wherein there is so much superabsorbent as to make the amount of wood pulp fiber appear relatively small in comparison.

Superabsorbent materials are also well known in the art. However, the present invention preferably employs a surface crosslinked particulate superabsorbent material. A presently preferred superabsorbent is a surface crosslinked particulate material available from the Hoechst-Celanese Corporation under the designation IM 2000.

The surface crosslinking of the particulate superabsorbent material desirably provides for the retention of the particulate shape when the superabsorbent material absorbs a liquid. Thus, the resulting hydrated superabsorbent (i.e., superabsorbent material that has absorbed a liquid) has a high gel strength.

It is believed that surface crosslinking plays an important role in helping to maintain the shape of the particulate superabsorbent material upon swelling when a liquid is absorbed. Surface crosslinking provides a more defined and ordered structure to the surface of the particulate superabsorbent material, and acts to restrain the superabsorbent material that is surrounded by the surface crosslinking from swelling in a disordered fashion.

When a superabsorbent particle of the present invention swell upon the absorption of a liquid, the crosslinked surface acts like an expandable net to restrain and order the swelling superabsorbent material.

The superabsorbent material may be described as comprising two regions: a central region that comprises a superabsorbent polymer and a surface region that comprises a superabsorbent polymer that is more highly crosslinked than the central region.

Preferably, the thickness of the surface region is less than about 10 percent of the total radial thickness of theoretical spherical superabsorbent particle.

It is also contemplated that other equivalent superabsorbent materials could be used in the present invention that would provide properties similar to a surface crosslinked superabsorbent particle. For example, a superabsorbent particle may be coated with a surface coating that acts to allow swelling of the superabsorbent material, but provides for maintenance of the shape of the particle upon swelling.

While not wishing to be bound by any particular theory, it is thought that gel blocking is ameliorated, despite the use a large quantity of superabsorbent material that forms a continuous phase, because rather that losing their shape, and therefore contacting each other to the exclusion of free volume, the crosslinked particulate superabsorbent material swells, but maintains a particular shape. Thus, as the superabsorbent particles swell, rather than coalescing with adjacent swollen particles, the particles contact and push against each other and thereby maintain free volume and a capillary structure or network through which liquid may be transported to that superabsorbent material that has not absorbed a liquid. In addition to maintaining

their shape, the volume of the section of the absorbent panel containing the continuous phase of superabsorbent particles increases markedly.

In addition, because the superabsorbent particles retain their shape and do not coalesce as a gel, the amount of free volume (as hereinafter defined) is not diminished greatly. Preferably, the free volume of the target region, at 600 seconds, during finite volume absorbency under load (FVAUL) testing is at least about 15 percent, and preferably at least about 20 percent, more preferably at least about 25 percent and most preferably at least about 30 percent. As noted, the target region 20 is generally located in the second and third fifths of the panel length, measured from the front of the diaper, and preferably comprises a top layer 22' predominantly of wood pulp fiber, a bottom layer 22' predominantly of wood pulp fiber and a middle layer that contains the continuous phase 21' of superabsorbent particles, in an amount ranging from about 70 percent to about 95 percent, preferably from about 80 percent to about 90 percent of the total weight of fibers and superabsorbent particles.

The present invention is also directed to a method and apparatus for calculating the FVAUL free volume of an absorbent composite comprising an SC-SAP material and wettable fibers. The present invention apparatus comprises a cylindrical open top holder for receiving a sample of the composite therein. A cylindrical weight having a screen secured at its bottom surface and a slot on one of its other surfaces is placed on top of the sample. The slot is in fluid communication with the screen at the bottom of the weight. As liquid is poured into the slot, it is evenly distributed through the screen on the top surface of the sample inside the holder. The apparatus further comprises means for holding the weight in place while allowing the weight to expand freely in a direction that is perpendicular to the top surface of the

sample upon absorbing the liquid. An LVDT device is operatively connected to said sample in order to measure the sample expansion. The LVDT device has rods that hold the weight in place on top of the sample. The holders, with the sample and the weight, are placed on top of a weight balance which measures the weight of the sample. The weight balance and LVDT devices are operatively connected to a computer. The data collected from the balance weight and the LVDT device are fed into the computer which calculates a free volume value at various time intervals from the time of the liquid addition.

Specifically, the method of calculating the free volume of the absorbent composite comprises placing a sample inside the holder, positioning a weight on top of the sample and pouring a liquid on the top surface of the sample through the weight slot. The liquid is evenly distributed on the top surface of the sample by the screen that is secured at the bottom of the weight. The sample is die-cut from the absorbent composite so that it has a cross section that matches the cross section of the holder and a volume about equal to the internal volume of the holder. The sample will thus tightly fit inside the sample holder. The method further consists of measuring the volume of the sample, measuring the mass of the sample using the weight balance, feeding said measured volume and mass values to a computer and calculating the free volume of the sample according to the equation.

$$FVS=VS- R*w/\rho SAPM -(1-R)*W/\rho PULP$$

wherein FVS is the free volume of the sample, VS is the volume of the sample, R is the weight ratio of SAP to sample weight, ρSAP is the density of the

SAP, ρpulpis the density of the pulp, and W is the mass of the sample and wherein R, ρSAP, and ρpulp are known values fed into the computer.

Examples:

Finite Volume Absorbency Under Load Method (FVAUL)

2" diameter samples of absorbent composites comprising wood pulp fiber and surface crosslinked superabsorbent material were die cut out of the cores of the absorbent article to be tested. The samples were equilibrated in a TAPPI conditioned room for 16 hours, and then placed in the holder 36 of the apparatus of Figure 3.

Figure 3 shows an apparatus used to measure finite volume absorbency under load (FVAUL), while Figure 4 shows a close up view of a weight 32 used in the FVAUL testing. The apparatus includes balance 34 and a sample holder 36 positioned on the balance, with the weight 32 configured for positioning on a test sample held by the sample holder. An LVDT (linear variable differential transducer) measuring system 38 is positioned to engage the weight 32 and measure its movement as a finite volume of liquid is introduced into the sample holder for absorption by a test sample. A Lucas Schaevitz Type 2000 HPA LVDT system was employed, which employed Lucas Schaevitz System 96 software. Since this software only provides LVDT measurements, additional software was provided to obtain readouts of values from balance 34, and of time.

As shown in Figure 4, the weight 32 includes a stainless steel tube 40 and a bottom stainless steel screen 42, with stainless steel slot 44 held within the tube and screen. Liquid to be introduced into a test sample is poured through the steel slot so that it passes through the screen 42 into the sample holder 36.

A computer software that can run the LVDT (linear variable differential transducer) system was booted. The LVDT system was calibrated, and the computer program to run the test was booted. 300 data sets were taken at two second intervals. A data set consists of time to the nearest hundredth second, balance reading to the nearest hundredth gram, and the LVDT reading to the nearest hundredth inch. The sample holder and a 0.16 psi porous weight were cleaned and then the holder was placed on a balance and the weight was put into place. The LVDT rods were then placed on the weights and the LVDT was zeroed.

The LVDT and the weight were removed and weighed and then the sample was placed into the holder (baby side up). The weight and LVDT were replaced and the computer program calculated the sample's thickness. The computer program asked for the sample weight and the ratio of superabsorbent particles (SAP) to sample weight. This information was used to determine the total volume being taken up by the SAP and pulp in the sample. The densities of 1.5 for SAP and 1.7 for pulp are used by the program. The computer the "calculates the free volume of the sample when dry. (If this value is known to be incorrect because of pad construction, it is possible to re-enter the free volume.)

An air shield was placed around the sample tester and the balance was zeroed (tared). 15 ml of test solution of 1 percent sodium chloride in water was prepared and placed in a graduated cylinder. The computer was then activated to start taking data sets and was allowed to take two data points before the solution was added. These two data sets are used to calculate the initial volume of the sample in the dry state. The 15 ml solution was quickly poured into the weight and was absorbed through the screen in the bottom of the weight into the sample. After the computer had taken 300

data sets, the computer generates the desired data such as dry free volume (the amount of air in the sample), the sample volume and sample mass as a function of time. The volume of the parts of the sample is calculated by taking the dry sample volume and subtracting the free volume from it and then adding the volume of liquid added.

$$\text{Volume parts} = (Vd - Vf) + L/1.01$$

Vd = Volume of Dry sample

Vf = free volume of air

L = weight of the liquid

1.01 = density of 1% NaCl solution

The sample volume and the volume by parts at 60 seconds and at 600 seconds was recorded.

The computer program that reads information from the LVDT system and the balance calculates the free volume for the dry sample and records that as the first record in the computer file. The calculation is based on three pieces of information: the sample weight, the ratio of superabsorbent to sample weight, and the sample thickness. The samples are all assumed to be two inches in diameter. The following equation shows how the calculation is done.

$$As = (2 \bullet 2.54.2)2 \bullet \pi =$$

$$Vs = As \bullet Ts$$

$$FVs = Vs - Vsap - Vpulp$$

$$Vsap = Msap/\rho sap$$

$$Vpulp = Mpulp/\rho pulp$$

Vs = Volume of the Sample (cm3)

As = Area of the Sample (cm2)

FVs = Free Volume of the Sample

Vsap = Volume of SAP in the Sample

Vpulp = Volume of Pulp in the sample

ρsap = Density of the SAP (g/(cm3)

ρpulp = Density of the pulp (g/(cm3)

$Msap = R \bullet W$ $Mpulp = (1-$

W = The mass of the Sample (g)

R = The ration of SAP to Sample

Ts = The thickness of the Sample (cm)

The following is the complete equation. 1.5 g/cc is used for the density of the superabsorbent 1.7 g/cc is used for the density of the pulp.

$FVs = 20.268 \bullet Ts - [R \bullet W/\rho sap] - [(1-R) \bullet W/\rho pulp]$

Tables 1 and 2 below represent the data obtained when the above described finite liquid volume absorbency under load protocol was run using a previously known layered absorbent structure, comprising IM 3900™ superabsorbent particles, and a layered structure embodying the present invention, comprising SC-SAP particles having identical performance characteristics as IM 4000™, designated by Hoechst-Celanese as S-347. In each test sample, the ratio of superabsorbent material to wood pulp fibers was 35:65. Each sample had three layers, including upper and lower layers predominantly of wood pulp fibers each at a basis weight of 150 g/m2; the resultant structures each had a layer of the superabsorbent particles in a substantially continuous phase, with the weight percentage of superabsorbent particles being 70 percent or greater. Each test sample was calendared to a thickness shown in the data. The columns are labeled to show sample volume (vol) and weight (wt) at 60 seconds (60 sec) and 600 seconds (600 sec) during testing after the addition of the liquid. The columns labeled 60 sec/percent free volume or air in the sample with respect to the total sample volume, thus representing the free volume of the tested sample.

The tested samples designated SCL are representative of the structure of the target region 20 of the absorbent structure 16 of the present absorbent article. While other portions of the absorbent structure preferably include less superabsorbent material, and may include less wood pulp fiber (by basis weight), practice of the present invention contemplates that at least the target region of an absorbent article, tested as specified above, exhibits a free volume, at 600 seconds, of at least about 15 percent, and preferably at least about 20 percent. A volume increase of at least about 20 percent, at 600 seconds, is also preferably exhibited.

Table 1

IM 3900, Layered 500/250 g/m3

		60 sec	60 sec	600 sec	600 sec	60 sec	600 sec	60 sec	600 sec
Test No.	Thickness (cm)	vol	wt	vol	wt	%Free Volume	% Free Volume	% Volume Increase	% Volume Increase
1	0.66	15.08	16.21	18.87	16.53	-7%	12%	7.5%	34.4%
2	0.70	16.92	16.26	17.83	16.23	4%	9%	13.2%	19.3%
3	0.73	15.40	16.43	16.64	17.40	-7%	-5%	-0.7%	7.3%
4	0.72	15.50	16.88	17.24	16.43	-9%	5%	0.9%	12.3%
5	0.71	15.41	16.51	17.07	16.42	-7%	4%	2.6%	13.6%
6	0.74	17.44	16.46	18.08	16.26	6%	10%	10.0%	14.1%
					Average	-3%	6%	5.6%	16.8%
					Stdev	6%	6%	5.5%	9.4%

Table 2

SCL SAP, Layered 500/250 g/m3

		60 sec	60 sec	600 sec	600 sec	60 sec	600 sec	60 sec	600 sec
Test No.	Thickness(cm)	vol	wt	vol	wt	%Free Volume	% Free Volume	% Volume Increase	% Volume Increase
1	0.69	15.77	15.06	19.65	15.15	5%	23%	7.1%	33.4%
2	0.72	18.78	16.61	22.19	16.44	12%	26%	21.9%	44.0%
3	0.67	16.00	15.89	19.95	15.87	1%	20%	11.9%	39.5%
4	0.71	14.81	15.93	18.47	15.74	-8%	15%	-1.6%	22.8%
5	0.69	71.77	16.05	21.32	15.72	10%	26%	20.7%	44.8%
					Average	4%	22%	12.0%	36.9
					Stdev	8%	5%	9.8%	9.1%

Note: Negative values of free volume reflect those test conditions during which all of the test solution had not yet been absorbed by the test specimen; actual free volume is approximately 0 percent.

Tables 3 and 4 below show the data obtained when the experiments represented in Tables 1 and 2 were replicated.

Table 3

SCL, Layered II

	60 sec	60 sec	600 sec	600 sec	60 sec	600 sec
Test No.	vol	wt	vol	wt	% Free Volume	% Free Volume
	14.06	16.15	17.62	16.06	-13%	10%
2	16.33	17.25	21.01	17.52	-5%	20%
3	15.68	15.61	20.20	15.52	0%	30%
				Average	-6%	20%
				Stdev	7%	10%

Table 4

IM 3900, Layered II

	60 sec	60 sec	600 sec	600 sec	60 sec	600 sec
Test No.	vol	wt	vol	wt	% Free Volume	% Free Volume
1	14.49	15.64	15.64	15.59	-7%	0%
2	14.41	15.37	15.54	15.14	-6%	3%
3	15.13	16.13	16.92	16.10	-6%	5%
				Average	-7%	3%
				Stdev	1%	2%

FIGURE 5 shows a graphical representation of the data obtained in Test No. 1 of Table 2.

FIGURE 6 shows a graphical representation of the data obtained in Test No. 5 of Table 1.

The data show that the increase of free volume is much greater when SCL superabsorbent particles are used in a layered structure than when IM 3900 superabsorbent particles are used in a layered structure. The sample volume refers to the total volume of the sample including free volume. The parts volume refers to the volume of the parts of the absorbent composite and include the wood pulp fibers, the SAP and the liquid.

What is claimed is:

1. An absorbent composite having a high fluid capacity without gel blocking upon absorption of liquid, said absorbent composite comprising wettable fibers and SC-SAP, wherein SC-SAP constitutes from about 70 percent to about 95 percent by weight based on the combined weight of the fibers and the SC-SAP, wherein said SC-SAP forms a substantially continuous phase and wherein the FVAUL free volume of the absorbent composite at 600 seconds is at least 15 percent.

2. The absorbent composite of claim 1 wherein said wettable fibers constitute at least 5 percent by weight based on the combined weight of fibers and the SC-SAP.

3. The absorbent composite of claim 1 wherein said wettable fibers are wood pulp fibers, meltblown synthetic fibers or mixtures thereof.

4. The absorbent composite of claim 1, wherein said FVAUL free volume at 600 seconds is from about 20 percent to about 40 percent.

5. The absorbent composite of claim 1, wherein said FVAUL free volume at 600 seconds is from about 25 percent to about 30 percent.

6. The absorbent composite of claim 5 wherein said SC-SAP constitutes from about 80 percent to about 95 percent by weight based on the combined weight of the fibers and the SC-SAP.

7. A disposable absorbent product comprising:

a liquid permeable top sheet;

a liquid impermeable back sheet; and,

an absorbent panel structure positioned intermediate said top sheet and said bottom sheet, said absorbent structure comprising a two phase absorbent matrix comprising wood pulp fiber and an SC-SAP wherein said SC-SAP forms a substantially continuous phase inside said absorbent panel, wherein said wood pulp fibers are dispersed in the interstices formed between said surface restructured SAP particles, and wherein said absorbent panel exhibits an FVAUL free volume of at least 15 percent at 600 seconds.

8. A disposable absorbent product in accordance with claim 7, wherein said absorbent panel further comprises a first layer comprising SC-SAP and wettable fibers wherein said SAP is nonuniformly distributed in said superabsorbent matrix in the Z-direction of said first layer.

9. A disposable absorbent product in accordance with claim 8, wherein said absorbent panel further comprises a second layer, adjacent said first layer with respect to the Z-direction of said absorbent panel, comprising predominantly wood pulp fibers.

10. A disposable absorbent product in accordance with claim 7, wherein said SC-SAP comprises at least 70 percent by weight based on the combined weight of the fibers and the SC-SAP.

11. A disposable absorbent product in accordance with claim 7, wherein said FVAUL free volume is from about 20 percent to about 30 percent at 600 seconds.

12. A disposable absorbent product comprising:

a liquid permeable top sheet;

a liquid impermeable back sheet; and,

an absorbent panel structure positioned intermediate said top sheet and aid bottom sheet, said absorbent structure comprising a two phase absorbent matrix comprising fibers and surface cross-linked polymeric superabsorbent material, wherein within at least a layer of said absorbent panel taken in the Z-direction thereof, said superabsorbent material comprises a substantially continuous phase of said matrix, within which a phase a sufficient quantity of particles of said superabsorbent material are in contact with each other to thereby define a capillary network for facilitating liquid transport within said panel structure wherein a target region of said absorbent structure exhibits an FVAUL free volume of from about 20 percent to about 30 percent at 600 seconds.

13. A disposable product in accordance with claim 12, wherein said fibers comprise wood pulp fibers.

14. A method of making an absorbent structure for use in a disposable absorbent, comprising the steps of:

providing a layer of wood pulp fibers;

distributing a layer of surface cross-linked superabsorbent material on said wood pulp layer;

providing another layer of wood pulp fibers on top of said layer of superabsorbent material; and,

calendaring said layers to form a two phase absorbent matrix wherein said layer of superabsorbent material comprises a substantially continuous

phase of said matrix, wherein a region of said absorbent matrix exhibits a free volume of from about 15 percent to about 30 percent at 600 seconds.

15. An apparatus for measuring the free volume of an absorbent composite comprising SAP comprising:

an open top holder for receiving a sample of said composite;

a weight having a screen at its bottom surface and a slot on one of its other surfaces said slot being in fluid communication with said screen wherein said weight has an outside diameter that is about equal to the inside diameter of said holder so that the weight will be resting on top of the sample;

means for holding the weight in place while allowing the weight to move freely in a direction that is perpendicular to the bottom surface of the weight to accommodate the expansion of the absorbent sample upon absorbing a liquid that is poured through the weight slot and the weight bottom screen on the sample inside the holder;

means for measuring the volume of the sample operatively connected to said sample; and,

balance means for placing said holder and measuring the weight of said sample.

16. The apparatus of claim 15, wherein said means for measuring the volume of the sample is an LVDT device.

17. The apparatus of claim 16, wherein said holding means are a set of rods operatively connecting said LVDT with said sample.

18. The apparatus of claim 17, wherein said balance means, and said LVDT device, are operatively connected to a computer for receiving the volume and weight measurements, calculating an FVAUL free volume value and displaying said FVAUL free volume value.

19. A method for calculating the free volume of an absorbent composite comprising a SAP material said method comprising the steps of:

providing an open top holder;

die-cutting a sample from said absorbent composite so that said sample has a cross section that matches the cross section of said holder and a volume equal or less to the internal volume of said holder so that the sample will completely fit inside said holder;

placing said sample inside said holder;

positioning a weight on top of said sample said weight having a porous screen attached at its bottom surface and a slot on one of its other surfaces said slot being in fluid communication with said porous screen;

pouring a liquid through said slot and through said screen into said holder to be absorbed by said sample causing the volume of the sample to increase thus displacing said weight;

measuring the volume of the sample using volume measuring means;

measuring the mass of the sample using weight means;

feeding said measured volume and mass values to a computer; and,

calculating the free volume of the sample according to the equation:

$$FVS=VS- R*w/\rho SAPM -(1-R)*W/\rho PULP$$

wherein FVS is the free volume of the sample, VS is the volume of the sample, R is the weight ratio of SAP to sample weight, ρSAP is the density of the SAP, ρpulpis the density of the pulp, and W is the mass of the sample and wherein R, ρSAP, and ρpulp are known values fed into the computer.

20. A method for calculating the free volume of an absorbent composite comprising a SAP material dispersed in a wood pulp fibrous material wherein the ratio of SAP to the total weight is R, the density of the SAP is ρSAP , the density of the pulp is ρpulp said method comprising the steps of:

providing an open top holder;

die-cutting a sample from said absorbent composite so that said sample has a cross section that matches the cross section of said holder and a volume equal or less to the internal volume of said holder so that the sample will completely fit inside said holder;

placing said sample inside said holder;

positioning a weight on top of said sample said weight having a porous screen attached at its bottom surface and a slot on one of its other surfaces said slot being in fluid communication with said porous screen;

pouring a liquid through said slot and through said screen into said holder to be absorbed by said sample causing the volume of the sample to increase thus displacing said weight;

measuring the volume of the sample using volume measuring means;

measuring the mass of the sample using weight means;

feeding said measured volume and mass values and said SAP ratio, and density values to a computer; and,

calculating the free volume of the sample using the computer according to the equation:

$$FVS=VS- R*w/\rho SAPM -(1-R)*W/\rho PULP$$

wherein FVS is the free volume of the sample, VS is the volume of the sample, is the weight ratio of SAP to sample weight, ρSAP= the density of the SAP, ρpulpis the density of the pulp, and W is the mass of the sample.

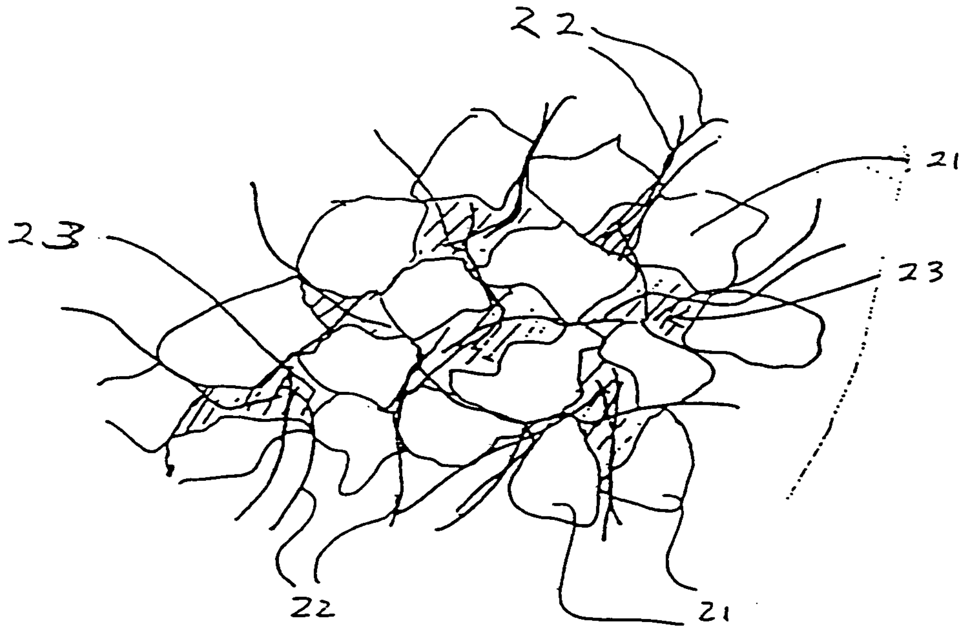


Figure 1

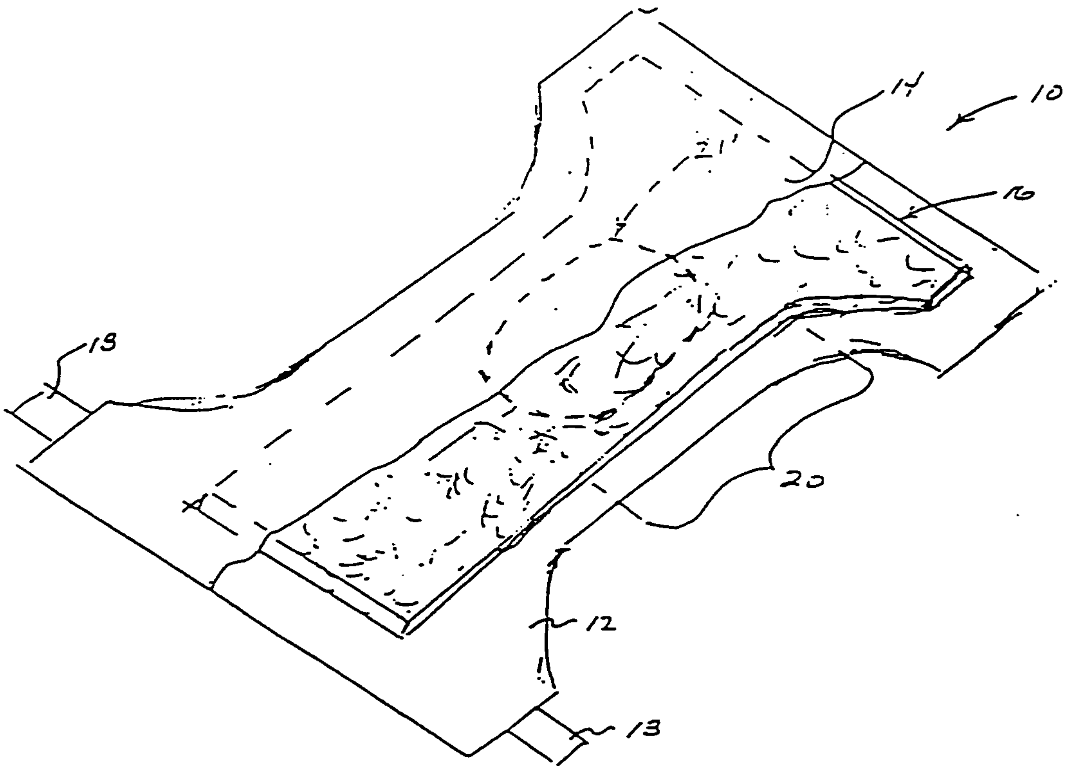


FIGURE 2

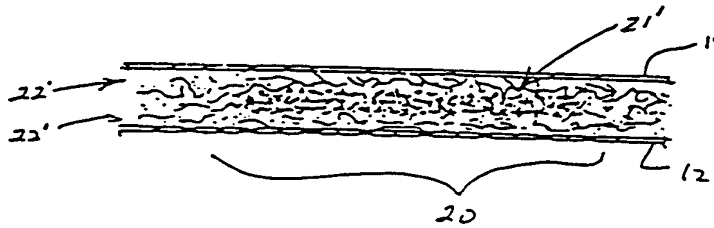


FIGURE 2a

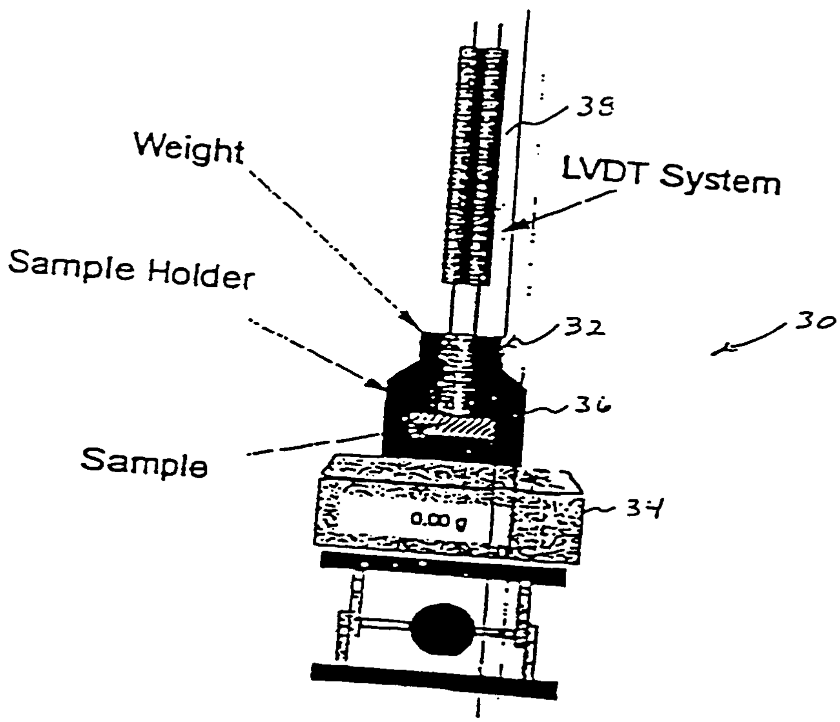


Figure 3

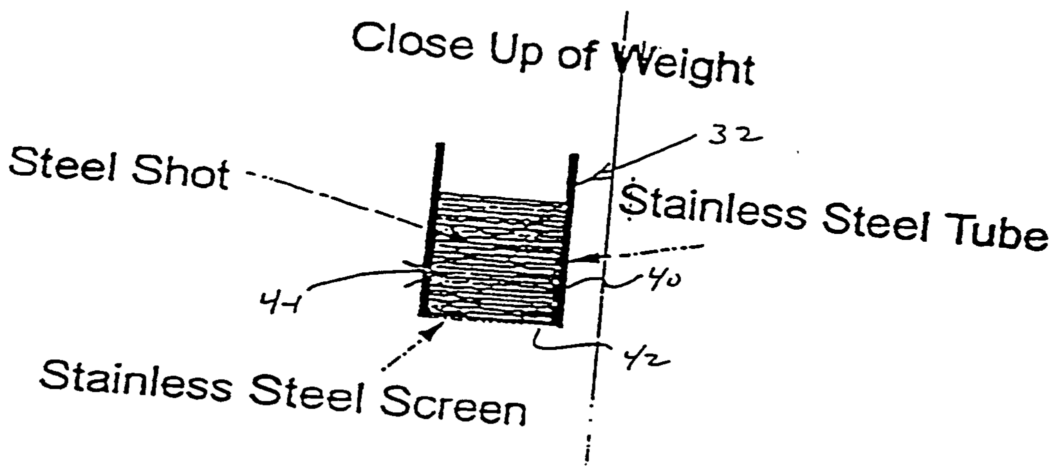

Close Up of Weight
Steel Shot
32
Stainless Steel Tube
40
44
42
Stainless Steel Screen

Figure 4

Figure 5

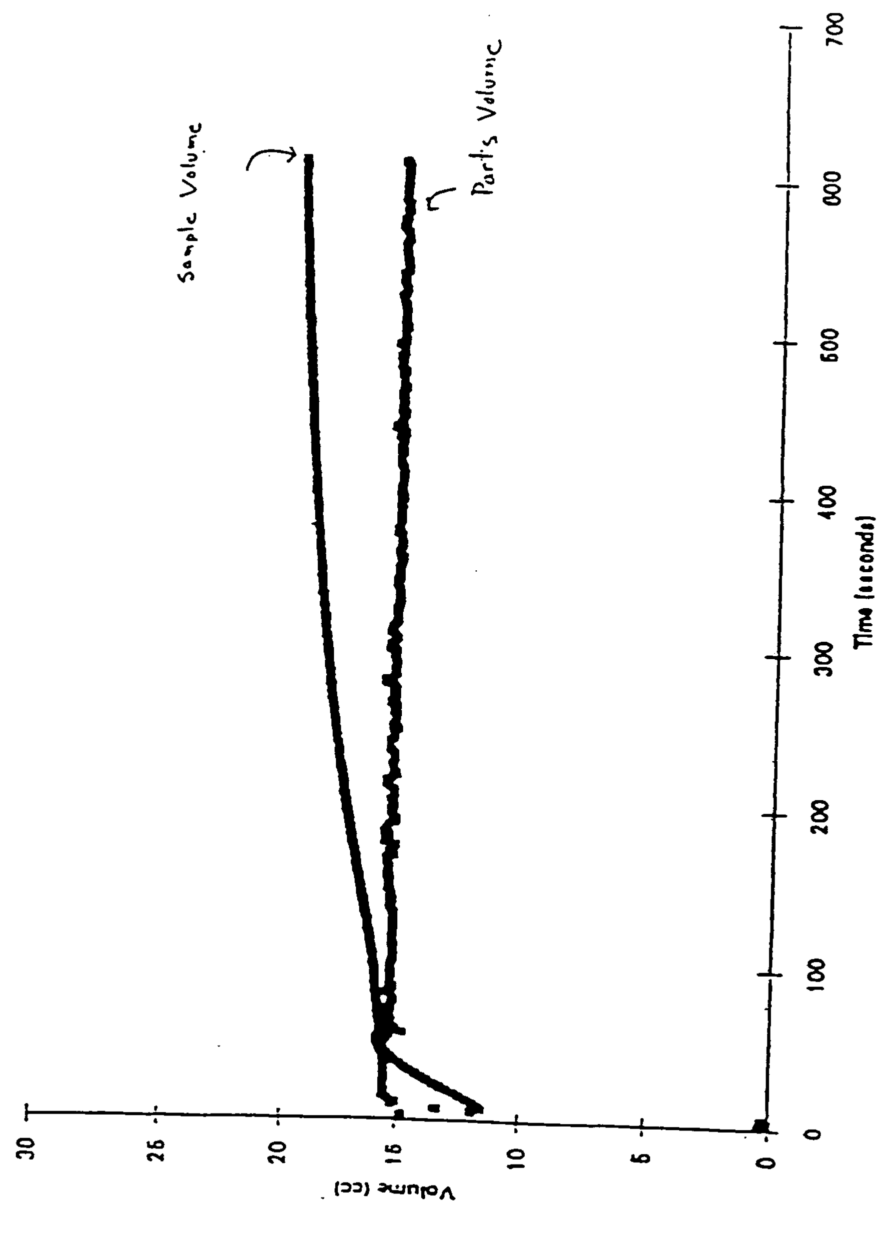
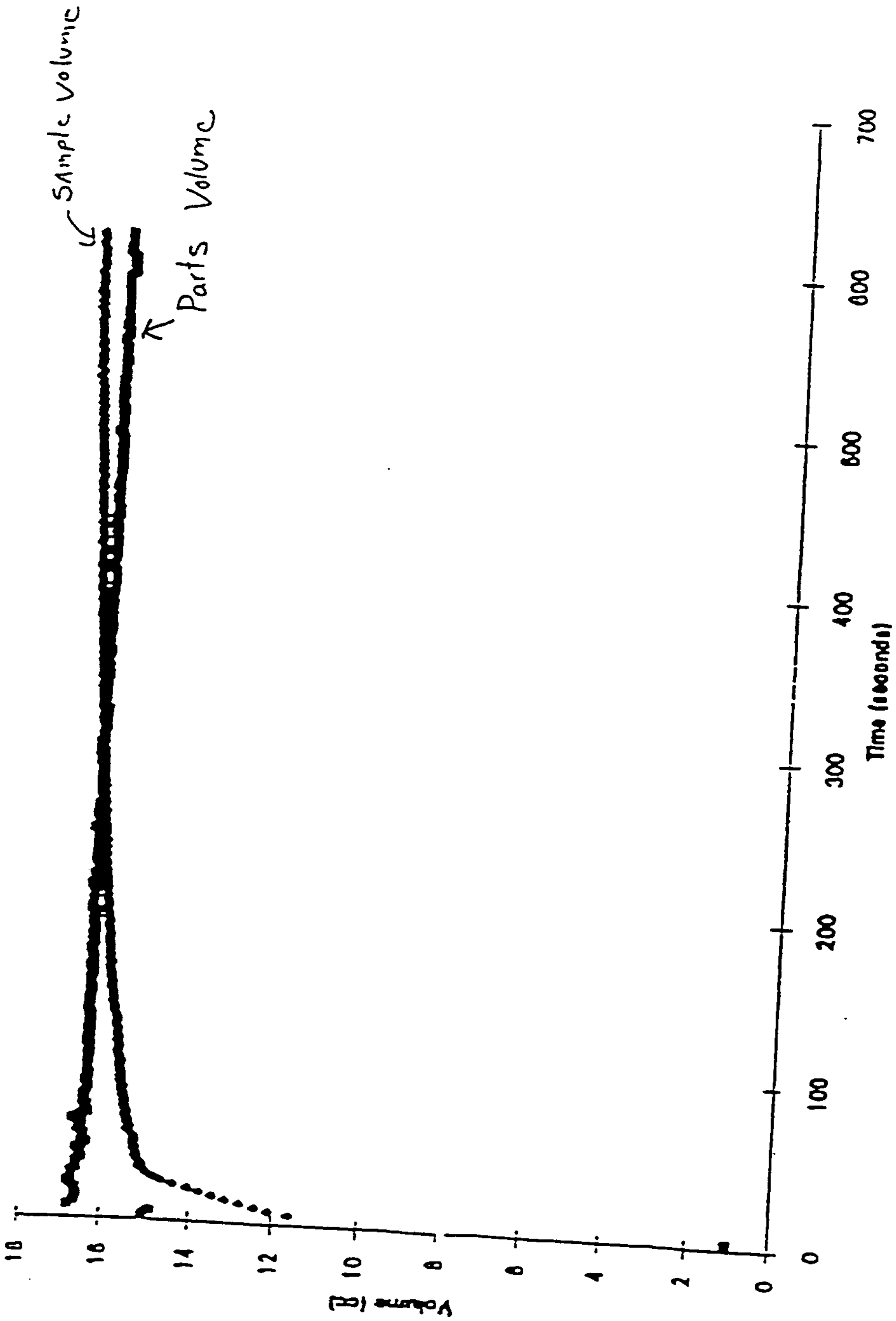

Sample Volume
Parts Volume
Volume (cc)
0
5
10
15
20
25
30
Time (seconds)
0
100
200
300
400
500
600
700

Figure 6


Sample Volume
Parts Volume
Time (seconds)
Volume (cc)
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300
400
500
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700
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6
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10
12
14
16
18

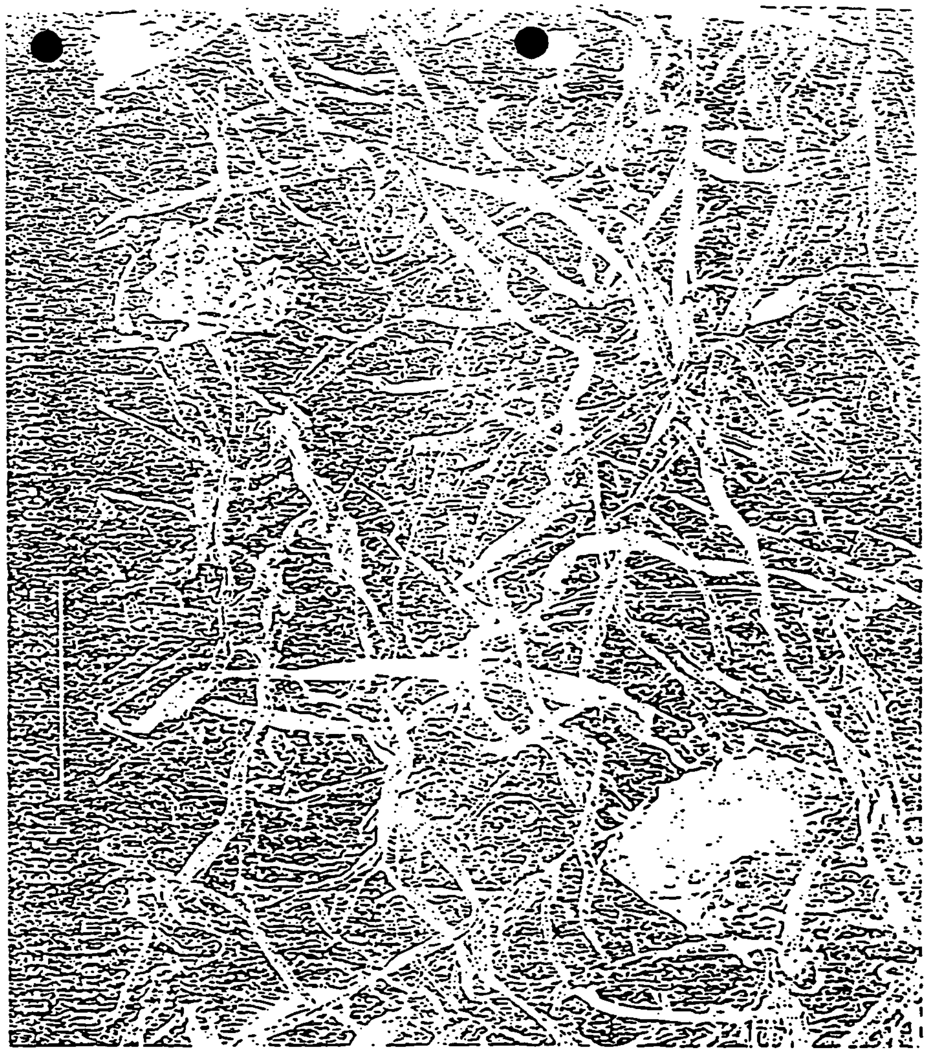

Figure 7

Figure 8

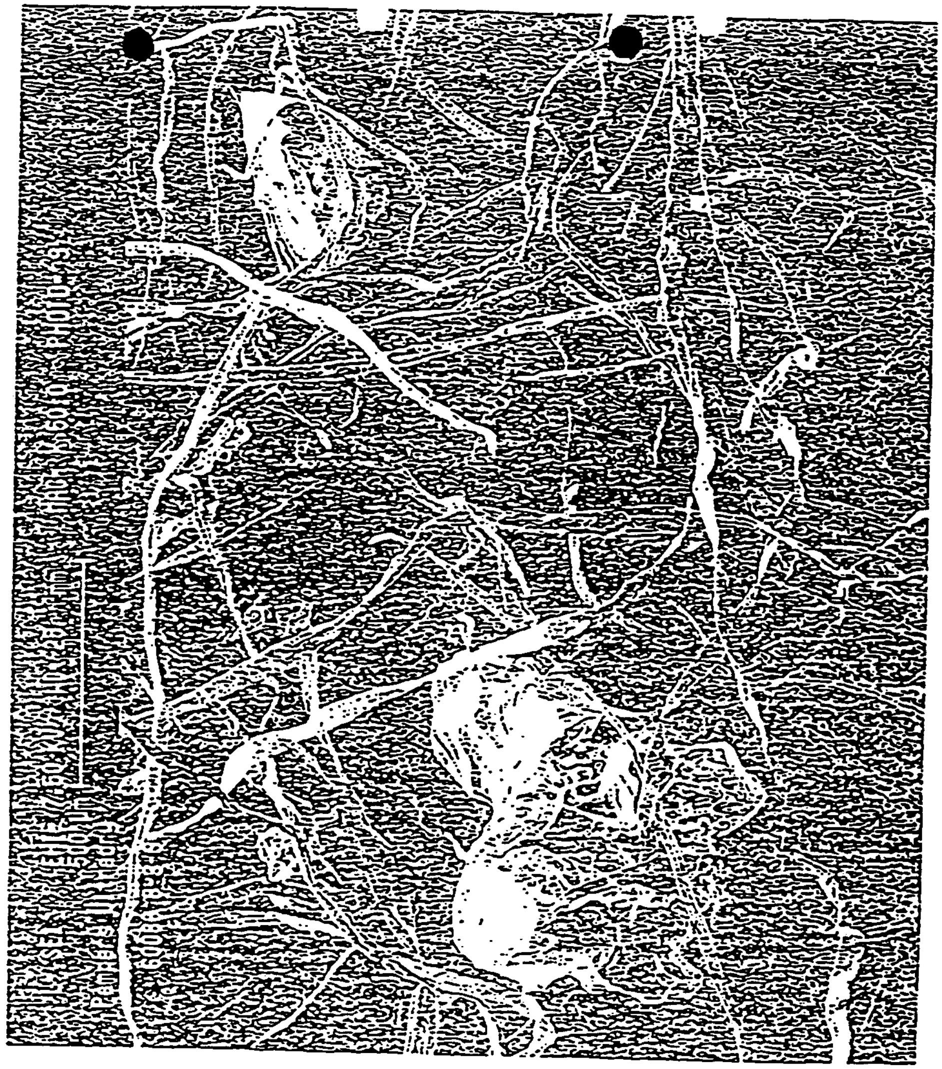

Figure 9

INTERNATIONAL SEARCH REPORT

International application No. PCT/US00/29338

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : A61F 13/15, 20
US CL : 604/367

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
U.S. : 604/367, 378, 385.01

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
APS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,505,718 A (ROE et al.) 09 April 1996, see entire document	1-14
A	US 5,137,600 A (BARNES et al.) 11 August 1992, see entire document	1-14
Y — A	US 5,675,079 A (GILMAN et al.) 07 October 1997, see entire document	15-19 — 20

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search
08 January 2001 (08.01.2001)

Date of mailing of the international search report
30 JAN 2001

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